RADIONUCLEAR MEASUREMENTS

FOR

QUALITY ASSURANCE

PROJECT PLAN

ASSESSMENT OF CHEMICAL CONTAMINANTS IN FISH CONSUMED BY FOUR NATIVE AMERICAN TRIBES IN THE COLUMBIA RIVER BASIN

Revision 1.1
July 31, 1997

Manager For Radionuclear Measurements: _____ Date: _____

Project Manager: _____ Date: _____

Project Risk Assessment Manager: _____ Date: _____

QA Officer: _____ Date: _____

ADDITIONAL SAMPLES FOR THE QUALITY ASSURANCE PROJECT PLAN FOR ASSESSMENT OF CHEMICAL CONTAMINANTS IN FISH CONSUMED BY FOUR NATIVE AMERICAN TRIBES IN THE COLUMBIA RIVER BASIN
REV. 6.0
12/16/97

ADDITION OF RADIONUCLIDE SAMPLE ANALYSIS

1.0 TASK DESCRIPTION

This work will support the Assessment of Chemical Contaminants in Fish Consumed by Four Native American Tribes in the Columbia River Basin by providing laboratory radiological analysis of fish samples collected from the Columbia River basin.

1.1 LABORATORY

Radionuclide analyses will be performed by the EPA National Air and Radiation Environmental Laboratory (NAREL) NAREL is a comprehensive environmental laboratory managed by the U.S. EPA Office of Radiation and Indoor Air. Among its responsibilities, NAREL includes a national program for collecting and analyzing environmental samples from a national network of monitoring stations for the analysis of radioactivity. This network, which has stations in every State, has been used to track environmental releases of radioactivity from nuclear weapons tests and nuclear accidents.

The NAREL radioanalytical program analyzes more than 10,000 samples annually and constitutes EPA's primary laboratory support for evaluation of ionizing radiation. Samples include air, water, soil, vegetation, human tissue, and food. NAREL applies quality assurance standards to all analyses, and routinely participates in laboratory intercomparison quality assurance programs with groups such as the World Health Organization and the International Atomic Energy Agency, as well as with the EPA Quality Assurance Program operated by the Agency's Office of Research and Development.

NAREL supports States in radiological environmental monitoring and has performed radiological surveys for the U.S. Navy. Through a cooperative agreement , NAREL is helping the Agency for Toxic Substances and Disease Registry to monitor radiation levels near major federal facilities.

1.2 TASK NARRATIVE

Samples will be shipped to the NAREL laboratory in Montgomery, AL. All samples will be analyzed by gamma spectrometry. Approximately 50% of all samples will also be analyzed for specific nuclides of uranium, thorium, plutonium, radium and strontium. Laboratory instruments

are maintained, calibrated, and operated in accordance with established NAREL procedures and SOPs.

NAREL quality assurance staff will assess laboratory operations in accordance with *The Quality Assurance Plan for the National Air and Radiation Environmental Laboratory* and written QA/QC policies for radionuclide analysis. Data packages and reports will be thoroughly reviewed in accordance with established NAREL procedures. Analytical data will be reviewed according to the *NAREL Standard Operating Procedure for the Review of Radioanalysis Data* (Draft, June 14, 1996).

This task will involve approximately 40-50 samples. Samples will be analyzed and reported within 24 weeks after the samples arrive at NAREL. A summary analytical report, including results of blanks and other QC samples and a case narrative describing sample exceptions, will be produced for each group of samples analyzed.

1.3 PERSONNEL, TRAINING, AND EQUIPMENT REQUIRED

NAREL personnel including staff from the Monitoring and Analytical Services Branch, and Quality Assurance will participate in each study. All laboratory analysts will be trained and certified for the analytical procedures they conduct at NAREL. NAREL has staff who are knowledgeable and who routinely perform data evaluation and interpretation.

NAREL maintains laboratory equipment used daily in the handling of radioactive samples. Equipment is maintained, calibrated, and used in accordance with NAREL SOPs and written policies and procedures.

2.0 ORGANIZATION AND RESPONSIBILITIES

According to the NAREL Quality Assurance Plan, the Director of the Office of Radiation and Indoor Air (ORIA) is responsible for ensuring that measurements performed within ORIA meet established Data Quality Objectives (DQOs). The Office Director has delegated the responsibility for overseeing quality assurance to the ORIA Quality Assurance Officer and has further delegated to the Director, NAREL, the primary responsibility for quality assurance on measurements in that facility. The Director, NAREL, has appointed a Quality Assurance Coordinator (QAC) to direct and oversee the laboratory's Quality Assurance Program. In addition, the Quality Assurance Forum (QAF) has been formed to focus on all pertinent QA issues. The QAF meets monthly, includes all interested laboratory personnel, and uses open forums and work groups to recommend procedures that will effectively and efficiently resolve an identified problem.

3.0 LABORATORY INSTRUMENTS AND EQUIPMENT

Laboratory instruments and equipment are used, calibrated, and maintained according to accepted good laboratory practices, written NAREL policies, and NAREL SOPs.

Regular efficiency checks are performed on every detector in use at the laboratory. Efficiency checks are performed daily for gas-flow proportional counters, scintillation counters, and germanium detectors. Checks are performed weekly for alpha spectrometers.

The results of efficiency checks are plotted on control charts and compared to warning and rejection limits. If an efficiency check fails on a detector, it is rerun once. If the second result is in the warning or rejection range, the detector is taken out of service and corrective action is initiated.

Regular background measurements are required for all detectors. Backgrounds are measured daily for proportional counters, twice a month for alpha spectrometers, monthly for germanium detectors, and immediately before a sample is counted on a scintillation counter.

Each gross radiation detector has an acceptable range for background levels. Background measurements are evaluated statistically to determine whether the true level is outside the acceptable range. There are warning limits and rejection limits for the test. If the most recent background level is outside the rejection limit, the detector is not used to analyze samples.

All radioactive standard solutions used for calibrations and efficiency checks at NAREL are traceable to the National Institute of Standards and Technology (NIST). Standard reference materials are purchased directly from NIST whenever they are available.

4.0 DOCUMENTATION AND RECORDS

4.1 NAREL DOCUMENT CONTROL SYSTEM

NAREL operates under a formal document control system, described in the *NAREL SOP for Document Control*, which presents the policies and procedures for the production, review, revision, storage, and distribution of documents. Document control policies apply to all printed internal documents that are maintained by or for NAREL personnel on a continuing basis for a period longer than one year. Controlled documents include, but are not limited to, the NAREL QMP, QAMs, QAPPs, SOPs, technical documentation, and forms. The Document Control Officer maintains the NAREL Document Control Logbook, maintains current copies of all controlled documents in hardcopy and electronic forms, approves any new or revised documents in the system, and has primary responsibility for the *NAREL SOP for Document Control.*

4.2 NAREL OPERATING DOCUMENTS

The *NAREL Quality Management Plan* (QMP) describes the Quality System at NAREL in terms of the organizational structure, functional responsibilities of management and staff, lines of authority, and processes for planning, implementing, documenting, and assessing activities. The QMP is the umbrella document for management policies, goals, and processes which incorporate Quality Assurance and Quality Control into all aspects of NAREL's work. The QMP describes how NAREL implements its Quality System and educates its staff about QA and QC processes.

A Quality Assurance Manual (QAM), formerly called a Quality Assurance Plan (QAP), presents technical criteria for analytical and administrative tasks to ensure that all data produced will be of known and desired quality, that all measurements performed at NAREL are valid, scientifically defensible, and of known precision and accuracy. The QAM addresses all phases of the quality control, quality assurance, and quality assessment processes. The manual presents specific and detailed information about tasks, processes, and criteria for various programs and activities. The QAM provides a detailed program for evaluating QC procedures and assessing results produced by the branch or program.

The Quality Assurance Project Plan, Assessment of Chemical Contaminants in Fish Consumed by Four Native American Tribes In the Columbia River Basin, Revision 6.0, December 16, 1996 provides specifications for required QA, QC, and reporting activities that must be implemented to ensure that the work performed on a specific project will satisfy the required performance criteria. Each project conducted by or for NAREL requires a QAPP. This includes projects supported by contract, interagency agreement, or grant. A QAPP is required to ensure that the analytical, sampling, and other programs meet the required DQOs that have been established for the project.

Standard Operating Procedures (SOPs) contain specific details and procedures which ensure that data generated by their use will be of known and adequate quality. All SOPs must be written, reviewed, approved, distributed, and revised in accordance with provisions in the *NAREL SOP for Writing SOPs.* An SOP details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps.

4.3 DATA PACKAGE DELIVERABLES

For each analytical batch of samples analyzed at the laboratory, a summary data package will be provided for each type of analysis. This summary includes:

- tabulated sample information: NAREL Sample ID, Client Sample ID, matrix, date collected, date received, and date analyzed.
- documentation exceptions
- holding time information if applicable
- sample preparation exceptions
- sample analytical exceptions
- general information unique to the sample batch, the analytical method, or reporting conventions
- individual report forms for each sample which provide
    - sample identification information
    - analytical method
    - detector identification
    - sample weight information
    - activity units
    - nuclides, activity, 2σ uncertainty, and MDC

4.4 RECORDS ARCHIVING AND RETENTION POLICIES

All records pertaining to environmentally related measurements will be archived, retained, and disposed of according to the pertinent EPA records schedule, with concurrence of the Navy and the NAREL project officer. Generally, hard copy records are maintained at NAREL for a minimum of ten years.

5.0 SAMPLE ANALYSIS

All samples will be analyzed for gamma-emitting nuclides with approximately 50% also analyzed for isotopic uranium, thorium, plutonium, radium and strontium. The NAREL Minimum Detectable Concentrations (MDC) for typical nuclides are listed in Tables 1 and 2. Sample size will be a minimum 600 g.

Target radionuclides for analysis were selected based on both on reviews of historical records for radionuclides previously detected or analyzed in the Columbia River and biota, and on the basis of current possible sources of radionuclides. Documents consulted included annual environmental monitoring reports from the Washington State Environmental Radiation Program and the Department of Energy Hanford Site Environmental Monitoring Program. Both short-lived and long-lived radionuclides were included consistent with ongoing reactor operations at the Washington Public Power Supply System nuclear plant as well as historic Hanford reactor operations.

Gamma analysis will be performed on all samples. The energy spectrum collected from the sample will be evaluated for gamma-emitting radionuclides over the energy range 60-2000 keV. Analysis will include naturally-occurring as well as manmade radionuclides. The analysis will quantify any spectrum peaks identified. In addition, minimum detectable concentrations for the following specified gamma emitting radionuclides will be quantified whether or not a peak is identified:

Be-7, Na-22, K-40, Mn-54, Co-58, Co-60, Fe-59, Zn-65,
Zr-95/Nb-95, Ru-103, Ru-106, Sb-125, I-131, Cs-134, Cs-137,
Ba-140/ La-140, Ce-141, Ce-144/Pr-144, Eu-152, Eu-154, Am-241

The following radionuclides will be analyzed in whole fish samples only. Strontium and plutonium, in particular, primarily accumulate in bone and should therefore be evaluated in whole fish rather than fillet.

Sr-89, Sr-90
Pu-238, Pu-239/Pu-240
U-238, U-234, Ra-226

Table 1. NAREL Minimum Detectable Concentration (MDC) for Selected Gamma Emitters Using Gamma Spectrometry with Ge Detector

Selected Gamma Emitters	MDC (pCi/L) for 1 L of Water Counted for 1000 min	MDC (pCi/gwet) for 1500 g of Sediment Counted for 1000 min	MDC (pCi/gwet) for 100 g of Sediment Counted for 1000 min	MDC (pCi/gwet) for 40 g of Sediment Counted for 1000 min	MDC (pCi/gwet) for 1000 g of Biota Counted for 1000 min
Am-241	17.7	0.0179	0.0842	0.168	0.0177
Cd-109	83.0	0.0749	0.424	0.901	0.0830
Th-234	52.5	0.0468	0.270	0.578	0.0525
U-235	56.7	0.0470	0.294	0.684	0.0567
Ra-226	86.1	0.0710	0.446	1.05	0.0861
Th-229	65.6	0.0540	0.340	0.799	0.0656
Pb-212	8.45	0.00689	0.0439	0.104	0.00845
Ra-224	91.0	0.0742	0.473	1.12	0.0910
Ra-223	26.7	0.0216	0.139	0.329	0.0267
Pb-214	11.4	0.00919	0.0601	0.143	0.0114
I-131	5.84	0.00468	0.0307	0.0730	0.0584
Rn-219	69.6	0.0556	0.367	0.873	0.0696
Be-7	45.4	0.0360	0.241	0.574	0.0454
Ba-140	22.2	0.0175	0.119	0.282	0.0222
Rn-220	7110	5.6	38.0	90.4	7.11
Tl-208	6.42	0.00504	0.0344	0.0818	0.00642
Cs-134	6.67	0.00523	0.0357	0.0850	0.00667
Bi-214	13.1	0.0103	0.0704	0.167	0.0131
Cs-137	7.26	0.00567	0.0391	0.0929	0.00726
Bi-212	89.4	0.0696	0.483	1.15	0.0894
Pb-211	188	0.145	1.02	2.42	0.188
Mn-54	7.03	0.00543	0.0382	0.0909	0.00703
Ra-228	24.4	0.0188	0.133	0.317	0.0244
Pa-234m	950	0.728	5.20	12.4	0.950
Co-60	10.3	0.00782	0.0566	0.135	0.0103
Na-22	9.38	0.00712	0.0519	0.123	0.00938
K-40	99.3	0.0749	0.552	1.31	0.0993

NOTE: MDCs will vary depending on activity in the sample, density of sample matrix, efficiency of detector, and other counting parameters. The above MDCs were calculated based on a 1000-min count of a 1.0-L Marinelli of deionized water.

Table 2. NAREL Minimum Detectable Concentration (MDC) for Selected Radionuclides Using Various Radiochemical Analyses

Radionuclide	*Matrix*	*Typical Aliquot Size*	*Count Time (min)*	*Method*	*MDC*
Gross Alpha	Water	250 mL	100	GFP	6 pCi/L
Gross Beta	Water	250 mL	100	GFP	3 pCi/L
Radium-226	Water	1 L	1000	SC	0.02 pCi/L
	Solids	0.5 g	1000	SC	0.04 pCi/g
Radium-228	Water	1 L	100	GFP	1 pCi/L
	Solids	0.5 g	100	GFP	2 pCi/g
Iodine-131	Water	2 L	1000	GFP	0.7 pCi/L
Uranium-234, 235, 238 Thorium-230, 232 Plutonium-238, 239	Water	1L	1000	AS	0.1 pCi/L
	Solids	0.5 g	1000	AS	0.2 pCi/g
Thorium-227	Water	1L	1000	AS	0.2 pCi/L
	Solids	0.5 g	1000	AS	0.35 pCi/g
Thorium-228	Water	1L	1000	AS	0.15 pCi/L
	Solids	0.5 g	1000	AS	0.3 pCi/g
Tritium	Water	10 mL	50	LS	400 pCi/L

AS Alpha Spectrometry
GFP Gas-Flow Proportional Counting
GS Gamma Spectrometry
LS Liquid Scintillation Counting
SC Scintillation Counting

6.0 SAMPLE LOCATIONS

Samples selected for radionuclide analysis are:

Site 7: sturgeon fillet without skin (3 replicates)

Site 8: sturgeon fillet without skin (3 replicates)
sturgeon whole (3 replicates)
whitefish fillet (3 replicates)
whitefish whole (3 replicates)

Site 9: sturgeon fillet without skin (3 replicates)
whitefish fillet (3 replicates)
whitefish whole (3 replicates)
other fillet (catfish or sucker) (3 replicates)
other whole (catfish or sucker) (3 replicates)

K Pond: sturgeon fillet without skin (3 replicates)
sturgeon whole (3 replicates)

Lab duplicates: 2

In addition: Samples from upstream (location 96)

sturgeon fillet without skin (3 replicates)
whitefish fillet (3 replicates)
whitefish whole (3 replicates)

7.0 SAMPLE MANAGEMENT

Environmental samples are received at NAREL, logged-in, and stored in accordance with the *NAREL SOP for Sample Receipt, Log-in, and Storage*. All samples are received by the Sample Preparation Manager (SPM) or designee. Sample coolers are stored in a secure area until they are surveyed for radioactive contamination. The results of the survey are recorded on the chain-of-custody forms.

The SPM compares the samples received to the chain-of-custody forms. Any discrepancies must be resolved and documented on the COC before sample analysis begins. The samples are then logged-in to the Sample Preparation Logbook and into the NAREL Radioanalytical Database.

After samples are logged-in and numbered, and all documentation is complete, samples are stored at various locations at NAREL, depending on the matrix, analyses requested, and project, until the analyses are performed.

Samples are always stored in a secure area to which only laboratory personnel have access. Access to NAREL is restricted. Outside entrances, laboratories, the counting room, and the sample preparation area require a key-card for entry. Visitors must sign in at the reception area and are escorted while in the laboratory.

The samples are shipped to NAREL where they are received by the NAREL sample preparation staff and transferred to the sample preparation laboratory. The packages are routinely screened, using beta and gamma detection equipment, to check external radiation levels before opening. The shipping containers are opened, the sample containers removed, and checked against the chain-of-custody documentation.

8.0 DATA MANAGEMENT

A sample identification code is assigned to each sample which is recorded in a logbook and entered into the NAREL database. Sample preparation activities are performed and the samples are distributed to analysts. Samples are subjected to appropriate analyses (alpha/beta screening, gamma spectroscopy, radiochemistry/ specific nuclides analysis), and the resulting data are subjected to verification by two independent parties. Following verification, the data are available in the database. At this point, a draft data package is prepared. The data package is reviewed and finalized by incorporating reviewers comments, as appropriate, and is then available for use by harbor studies personnel.

A draft of the *NAREL Standard Operating Procedure for the Review of Radioanalysis Data* includes:

* sample receipt and preparation,
* laboratory data handling (using the Laboratory Information Management System (LIMS),
* instrumentation calibrations and efficiency checks,
* types of sample analyses performed (individual detailed procedures are available for each type),
* data review, error detection and other problem determinations, including requirements for recounting or reanalysis of samples,
* involvement of the project quality assurance officer and the NAREL quality assurance coordinator,

Record keeping is in the form of laboratory logbooks and information and data storage in the NAREL LIMS system (which includes a large database of storage of all data produced in the radioanalytical laboratory). NAREL has a formal document control system which allows for documents to be either controlled or uncontrolled. The procedures used to administer this system

are included in the procedure entitled *NAREL Standard Operating Procedure for Document Control*. Data storage and retrieval is via the LIMS system referenced above.

The methods for detecting and correcting errors are used in NAREL data management activities including verification of samples with chain-of-custody records, instrument calibration and background checks, and extensive review of counting room data before release. Loss of data during entry, reporting or reduction is very unlikely since data are maintained on a local area network where nightly backups are performed, and data are archived to optical disks on a regular basis. Any calculations requiring the data extract a copy from the database, leaving the original electronic record intact. The only chance for temporary data loss is in case of a power failure during sample counting or hardware failure. In those instances, the sample is still available and is simply recounted.

Data handling equipment includes radioanalytical instrumentation, PC's dedicated to counting room applications, and the NAREL local area network. All of this equipment has been thoroughly tested for these applications and proven to be stable and reliable. Commercial software currently used by the radioanalytical laboratory is as listed below:

Program	Analysis System
LB4000	Tennelec LB4000 Gas-Flow Proportional Counters (raw data)
GDR	High-purity Germanium Detectors (raw and reduced data)
AlphaMat	Alpha Spectrometers (raw and reduced data)
G3000	Gamma Products G3000 Automatic Germanium Counting System (control software)
G5000	Gamma Products G5000 Automatic Alpha/Beta Counting System (control software)

In-house software includes the following:

Data Entry and Instrument Control

Program	Analysis System
I131	Tennelec LB4000 Iodine-131
GROSS	Tennelec LB4000 Gross alpha and beta
SR	Tennelec LB4000 Strontium-89 and 90
RA228	Tennelec LB4000 Radium-228
TH234	Tennelec LB4000 Thorium beta tracer
GET4, GET12	Germanium counters

Calculation and Data Review

Program	Analysis System
GAMMARVW	Gamma spectrometry

ALPHARVW	Alpha spectrometry (Am,U,Pu,Th)
I131RVW	LB4000 Iodine-131
GROSSRVW	LB4000 Gross alpha and beta
SRRVW	LB4000 strontium-89 and 90
RA226	Radium-226 by the radon emanation method

Data Management

CAA	NAREL database system
RPT	Interactive database queries
NARSS	Counting Room analysis scheduling
DATAPKG	Data package production

The calculations performed by all in-house analysis software are documented in the software user manuals. Only one NAREL employee is authorized to modify in-house analysis software and electronic and written records of software modifications are maintained. After each modification of an analysis software system, the calculations are checked using a calculator program, which reads equations from a text file in a form similar to that shown in the user's manual. The results generated by the analysis software are checked against the results given by the calculator.